

Data Validation SOP

HW-22, Rev. 2

Semivolatile Organics

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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8270C" December, 1996. Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 4.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications

Data reviewers must possess a working knowledge of SW846 Analytical Method 8270C.

YES NO N/A

DEFINITIONS

Acronyms

BNA - base neutral acid (another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB - decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
l - liter
ml - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCX - tetrachloro-m-xylene

YES NO N/A

TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

YES NO N/A

Data Qualifiers

- U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
- NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

YES NO N/A

LAB QUALIFIERS:

- D - The positive value is the result of an analysis at a secondary dilution factor.
- B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.
- E - The concentration of this analyte exceeds the calibration range of the instrument.
- A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.
- X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

YES NO N/A

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____ LAB: _____

SITE NAME: _____

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable
format? [] _____

ACTION: If not, note the effect on review of the data
in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter
present? [] _____

2.2 Are case number and SDG number(s) contained
in the narrative or cover letter? [] _____

YES NO N/A

II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples?

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any semi volatile technical holding times, determined from date of collection to date of extraction, been exceeded?

Continuous extraction of water samples for semi volatile analysis must be started within 7 days of the date of collection. Soil/sediment samples must be extracted within 14 days of collection. Extracts must be analyzed within 40 days of the date of extraction.

YES NO N/A

Table of Holding Time Violations

Sample ID	Sample Matrix	Date Sampled	(See Traffic Report)		
			Date Lab Received	Date Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

YES NO N/A

3.0 Surrogate Recovery (Form II)

3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:

a. Low Water

b. Low/Med Soil

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:

a. Low Water

b. Low/Med Soil

ACTION: If CLP deliverables or equivalent are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk?
ACTION: Circle all outliers in red.

YES NO N/A

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank ____ []

If yes, were samples re-analyzed? []

Were method blanks re-analyzed? []

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, for the affected fraction only (i.e. either base-neutral or acid compounds):

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral or acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".
2. Non-detects for that fraction should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses.

YES NO N/A

3.5 Are there any transcription/calculation errors
between raw data and Form II? []

ACTION: If large errors exist, call lab for
explanation/resubmittal, make any
necessary corrections and document
effect in data assessments.

4.0 Matrix Spikes (Form III)

4.1 Have the semivolatile Matrix Spike and
Matrix Spike Duplicate or duplicate unspiked
Sample recoveries been listed on the CLP
Recovery Form (Form III)? []

NOTE: This method may not require a Matrix Spike
Duplicate. Lab should submit MS/MSD or MS and
Duplicate unspiked sample. (see section 8.4.2,
page 8270C-22)

4.2 Were matrix spikes analyzed at the required
frequency for each of the following matrices:

a. Low Water	<u>[]</u>	<u> </u>
b. Low Solid	<u>[]</u>	<u> </u>
c. Med Solid	<u>[]</u>	<u> </u>

ACTION: If any matrix spike data are missing, take
the action specified in 3.2 above. It may be
necessary to contact the lab to obtain the
required data.

YES NO N/A

NOTE: If the data has not been reported on CLP forms, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

- 4.3 Were matrix spikes performed at concentration > 100ug/L ? ☐ ☐ ☐
- 4.4 Were any semivolatile spike recoveries outside QC limits (compare to the values in Table 6, page 8270C-39 and 40) or Lab's in-house generated criteria? ☐ ☐ ☐
- 4.5 Were any RPD's for matrix spike and matrix spike duplicate recoveries outside QC limits? ☐ ☐ ☐

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using professional judgement, the data reviewer may use the matrix spike / matrix spike duplicate and duplicate unspiked results in conjunction with other QC criteria to determine the need for some qualification of the data.

YES NO N/A

4.6 Was a LCS analyzed with each analytical batch? (See section 8.4.3, page 8270C-22)

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

4.7 Were any LCS recovery outside the interim acceptance criteria of 70 - 130% or outside lab's in-house generated limits?

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present?

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch?

5.3 Has a method blank been analyzed for each GC/MS system used ?

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

YES NO N/A

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

YES NO N/A

6.2 Do any field/rinse/ blanks have positive results
for target analytes and/or TICs (if required,
see paragraph 10 below)? []

ACTION: Prepare a list of the samples associated
with each of the contaminated blanks.
(Attach a separate sheet.)

NOTE: All field blank results associated to a
particular group of samples (may exceed one
per case) must be used to qualify data.
Blanks may not be qualified because of
contamination in another blank. Field blanks
must be qualified for outlying surrogates,
poor spectra, instrument performance or
calibration QC problems.

ACTION: Follow the directions in the table below to
qualify sample results due to contamination.
Use the largest value from all the
associated blanks. If gross contamination
exists, all data in the associated samples
should be qualified as unusable (R).

**For Common Phthalate
Esters:**

Sample conc. > CRDL,
but < 10x blank
result

Sample conc. is
< CRDL & < 10x blank
result

Sample conc. > CRDL &
> 10x blank result

**For Other
Contaminants:**

Sample conc. > CRDL,
but < 5x blank result

Sample conc. < CRDL &
< 5x blank result

Sample conc. > CRDL &
> 5x blank result

Action:

Flag sample result
with a "U"

Report CRDL and
qualify with a "U"

No qualification is
necessary

YES NO N/A

NOTE: Analytes qualified "U" for blank contamination are still considered as hits when qualifying for calibration criteria.

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required. (see section 7.6.2, page 8270C-19)

6.3 Are there field/rinse/equipment blanks associated with every sample? ☐

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Apparatus and Materials

7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270C? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column. ☐

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

YES NO N/A

8.0 GC/MS Instrument Performance Check

8.1 Are the GC/MS Instrument Performance Check Forms
(Form V) present for decafluorotriphenylphosphine
(DFTPP)?

NOTE: The performance solution should also contain 4,4-DDT,
pentachlorophenol, and benzidine to verify
injection port inertness and column performance.
The degradation of DDT to DDE and DDD must be
less than 20% total and the response of
pentachlorophenol and benzidine should be
within normal ranges for these compounds (based
upon lab experience) and show no peak degradation
or tailing before samples are analyzed. (see section 5.5
page 8270C-11).

8.2 Are the enhanced bar graph spectrum and
mass/charge (m/z) listing for the DFTPP
provided for each twelve hour shift?

8.3 Has an instrument performance check solution
been analyzed for every twelve hours of sample
analysis per instrument?

ACTION: List date, time, instrument ID, and sample
analyses for which no associated GC/MS
tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

YES NO N/A

ACTION: If the lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198? []

8.5 Have the ion abundance criteria been met for each instrument used? []

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, the Region II TOPO must be notified.

8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) []

8.7 Have the appropriate number of significant figures (two) been reported? []

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

8.8 Are the spectra of the mass calibration compound acceptable? []

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

YES NO N/A

9.0 Target Analytes

9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:

- a. Samples and/or fractions as appropriate ☐ ☐
- b. Matrix spikes and matrix spike duplicates ☐ ☐
- c. Blanks ☐ ☐
- D. Lab control samples ☐ ☐

9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270C-13)? ☐ ☐

ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.

9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

- a. Samples and/or fractions as appropriate ☐ ☐
- b. Matrix spikes and matrix spike duplicates (Mass spectra not required) ☐ ☐
- c. Blanks ☐ ☐
- D. Lab control samples ☐ ☐

ACTION: If any data are missing, take action specified in 3.2 above.

YES NO N/A

9.4 Is chromatographic performance acceptable with respect to:

Baseline stability?	<input type="checkbox"/>	___
Resolution?	<input type="checkbox"/>	___
Peak shape?	<input type="checkbox"/>	___
Full-scale graph (attenuation)?	<input type="checkbox"/>	___
Other: _____	<input type="checkbox"/>	___

ACTION: Use professional judgement to determine the acceptability of the data.

9.5 Are the lab-generated standard mass spectra of identified semivolatile compounds present for each sample? ☐ ___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.6 Is the RRT of each reported compound within ± 0.06 RRT units of the standard RRT in the continuing calibration? ☐ ___

9.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? ☐ ___

YES NO N/A

9.8 Do the relative intensities of the characteristic ions in the sample agree within $\pm 30\%$ of the corresponding relative intensities in the reference spectrum? [] ____

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected ("U") at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form I's, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? [] ____

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: [] ____

a. Samples and/or fractions as appropriate [] ____

b. Blanks [] ____

ACTION: If any TIC data are missing, take action specified in 3.2 above.

YES NO N/A

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)? []

- ACTION: i. Flag with "R" any target compound listed as a TIC.
- ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? []

10.5 Do TIC and "best match" standard relative ion intensities agree within $\pm 20\%$? []

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R".

YES NO N/A

11.0 Compound Quantitation and Reported Detection Limits

NOTE: **Average Response Factor from the initial calibration is used for quantitation.**

11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? []

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two co-eluting peaks to calculate the total concentration).

11.2 Are the method detection limits adjusted to reflect sample dilutions and % moisture in case of soil samples? []

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

YES NO N/A

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration?

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI)

13.1 Are the Initial Calibration Forms (Form VI) present and complete for the semivolatile fraction?

ACTION: If any calibration forms or standard raw data are missing, take action specified in 3.2 above.

YES NO N/A

13.2 Are all **average RRF's** of the four System Performance Check Compounds (**SPCCs**): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol and 4-nitrophenol > 0.050?

ACTION: If no:
CONTRACTUAL - Circle all outliers in red.
Document in the Data Assessment under contract non compliance.

ACTION: TECHNICAL - For any target analyte, CCC or SPCC with average **RRF <0.05**

1. "R" all non-detects;
2. "J" all positive results.

13.3 Is the % RSD for each individual Calibration Check Compound (CCC) - Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, Diphenylamine, Di-n-octyl phthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol and 2,4,6-Trichlorophenol less than 30%?

ACTION: If no:
CONTRACTUAL - Circle all outliers in red.
Document in the Data Assessment under contract non compliance.

TECHNICAL - All positive hits for that particular CCC must be qualified "J". If % RSD > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.

YES NO N/A

13.4 If % RSD for one or more target analytes exceeds 15%,
is the MEAN of the % RSD values for ALL analytes in
the calibration less than or equal to 15%?

ACTION: If yes:
CONTRACTUAL - The initial calibration is
valid and the average RF from the initial
calibration is used to quantitate sample results.

TECHNICAL - If the % RSD is \geq 15.0% for any
Individual target analyte, qualify positive
results for that analyte "J". If % RSD > 90%,
flag all positive results for that analyte "J" and
non-detect results for that analyte "R" unusable.

13.5 If the MEAN % RSD is greater than 15%, Did the
laboratory calculate first or second order
regression fit of the calibration curve ?

ACTION: If no:
CONTRACTUAL - The initial calibration is
not valid, Document in the Data Assessment
under contract non compliance.

TECHNICAL - If % RSD is > 15.0% for any
individual target analyte, qualify positive
results for that analyte "J". When % RSD > 90%,
flag all positive results for that analyte "J"
and non-detect results for that analyte "R"
unusable.

NOTE: Analytes previously qualified "U" due to
blank contamination are still considered
as "hits" when qualifying for calibration
criteria.

YES NO N/A

13.6 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.)

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

13.7 Do the target compounds for this SDG include Pesticides?

13.8 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?

ACTION: If DDT percent breakdown exceeds 20%:

- i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".
- ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

YES NO N/A

14.0 GC/MS Calibration Verification (Form VII)

14.1 Are the Calibration verification Forms (Form VII) present and complete for all compounds of interest?

14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

ACTION: If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If calibration verification data is not available, flag all associated sample data as unusable ("R").

	YES	NO	N/A
14.3 Do any of the SPCCs have an RRF <0.05 ?	___	<u>[]</u>	___
If YES, did the lab take corrective action as in section 7.4.4.2, page 8270C-17.	<u>[]</u>	___	
ACTION: If no: CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.			
ACTION: TECHNICAL - For any target analyte, SPCC or CCC with RRF <0.05			
1. "R" all non-detects;			
2. "J" all positive results.			
14.4 Do any of the CCCs have a %D between the initial and verification RRF which exceeds 20.0%?	___	<u>[]</u>	___
ACTION: If Yes: CONTRACTUAL - Circle all outliers in red. Document in the Data Assessment under contract non compliance.			
TECHNICAL - All positive hits for that particular CCC must be qualified "J" and all non-detects "UJ". When %D > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.			
14.5 Do any target compounds have a % D between the initial and verification RRF which exceeds 20.0%?	___	<u>[]</u>	
ACTION: If yes: CONTRACTUAL - Circle all outliers in red.			
TECHNICAL - All positive hits for that particular target compound must be qualified "J" and all non-detects "UJ". When %D > 90%, flag all positive results for that analyte "J" and non-detect results for that analyte "R" unusable.			

YES NO N/A

14.6 If %D for one or more target analytes exceeds 20%,
Is the MEAN of the %D values for all analytes in
The calibration less than or equal to 20%?

ACTION: If yes:
CONTRACTUAL - The initial calibration is
valid and the average RF from the initial
calibration is used to quantitate sample results.

If no:
The initial calibration is invalid. Document
in the Data Assessment under contract non compliance.

14.7 Are there any transcription/calculation errors in
the reporting of average response factors (RRF) or
percent difference (%D) between initial and
continuing RRFs? (Check at least two values but if
errors are found, check more).

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for
explanation/resubmittal, make any necessary
corrections and document effect(s) in the
data assessments.

YES NO N/A

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration?

[] [] []

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area	LowerLimit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

- ACTION:
- i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.
 - ii. Non-detects associated with IS > 100% should not be qualified.
 - iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).

YES NO N/A

15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard? ☐ ☐ _____

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Field Duplicates

16.1 Were any field duplicates submitted for semivolatile analysis? ☐ ☐ _____

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.